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PROGRESS REPORT

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MECHANICAL BEHAVIOR OF POLYCRYSTALLINE NON-METALLICS AT ELEVATED TEMPERATURE

for the period

October 1, 1965 through March 31, 1966

submitted to

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National Aeronautics and Space Administration

Washington, D. C. 20546

submitted by

Department of Materials Science
Stanford University
Stanford, California

Professor Oleg D. Sherby, Principal Investigator
Mr. Peter Burke, Research Assistant

MECHANICAL BEHAVIOR OF POLYCRYSTALLINE NON-METALLICS
AT ELEVATED TEMPERATURES
(SC-NGR-05-020-084)

Summary of Research for the Period October 1, 1965 Through March 31, 1966

During the last six months, research efforts have been concentrated primarily on creep of sodium chloride. Polycrystalline samples have been made by extruding single crystals. These samples have been tested in compression creep at temperatures of 512 and 740°C and stress levels varying from 38 to 850 psi. The results of these tests indicate that the steady state creep rate, $\dot{\epsilon}$, can be expressed by an equation which has been used to describe dislocation climb and glide controlled creep in metals, namely,

$$\dot{\epsilon} = Ae^{-Qc/RT}\sigma^{n}$$

where $Qc \simeq 73$ Kcal/mole = 3.2eV = the activation energy for creep,
$n \simeq 7$ = the stress exponent,
R = gas constant,
T = absolute temperature
and σ = creep stress.

For metals, $n \simeq 5$, and Qc equals the activation energy for diffusion, Qd, plus factors dependent upon stacking fault energy and elastic modulus changes with temperature according to the following equation (7);

$$Qd = Qc - nR\frac{d\ \ln E}{d\ 1/T} - 3.5R\frac{d\ \ln \gamma}{d\ 1/T}$$

where E = elastic modulus
and γ = stacking fault energy

A value for Qd for sodium chloride can be calculated from the above equation by using elastic modulus values extrapolated from available data (9) and assuming that the change in stacking fault energy with temperature is negligible. The calculated value of Qd $\leqslant$61 Kcal/mole agrees fairly well with observed activation energies for chlorine diffusion in sodium chloride, which range from 45 to 54 Kcal/mole (1,2). The activation energy for sodium diffusion in sodium chloride is 37 Kcal/mole (1). The difference between the observed and calculated values of Qd could be a real difference due to differences in the compositions and histories of the creep and diffusion samples, or it

could be due to experimental error, error in the extrapolated modulus values, changes in stacking fault energy with temperature, or effects due to grain growth and grain size differences between the creep samples.

Microstructures developing during creep of sodium chloride were found to be similar to those developing in metals undergoing creep deformation. In both cases subgrains form during creep, and the subgrain size is inversely proportional to the creep stress. Grain growth occurred, but for metals it has been found that the grain size has little effect on either the subgrain size or the creep rate (10).

From the above results it seems likely that creep in sodium chloride is similar to creep in pure metals from both a kinetic and a microstructural point of view.

Creep testing of aluminum oxide has not begun, but it is hoped that the equipment for these tests will be operative in about three months. The equipment will also be used for high strain rate ($> 10^{-3} sec^{-1}$) creep testing of sodium chloride.

The project now has a laboratory of its own that has become available as the materials science facilities of Stanford have grown. Most of the equipment pertinent to the project has been moved into it. The laboratory is well equipped with power and water, including a 30 KVA transformer, which will be necessary for heating a silicon carbide furnace to be used for aluminum oxide testing.

A report is currently under preparation by Oleg D. Sherby and Peter M. Burke on the elevated temperature behavior of polycrystalline metals and non-metallics. This report will critically review the factors influencing creep behavior of crystalline solids at elevated temperature. Special emphasis will be placed on a comparison of the dissimilarities and similarities of creep for metallics and non-metallics. The paper will be submitted as a technical report to NASA and should be completed by the end of June.

<u>Creep Testing of Sodium Chloride</u>

<u>Sample Preparation</u>

Polycrystalline samples of 0.003 mm grain size have been made by extruding a single crystal grown from reagent grade sodium chloride.

The extrusion dies are made of hardened steel. The reduction ratio is 16:1, and the sample diameter is 1/4 inch. Extrusion temperatures ranged from 250 to 350^{o}C, and extrusion forces ranged from 17,000 to 35,000 pounds.

The extruded samples were glued to a piece of lucite for support and sawed approximately to length with a jeweler's saw. The glue and plastic were dissolved in acetone. The rough cut samples were then glued to a metal jig and polished so that the ends were flat and parallel and normal to the sample cylindrical axis.

Density was determined by measuring the sample dimensions and weight and calculating the density. Densities measured were 98 to 99% of theoretical density.

Creep Testing

Creep tests were conducted using a dead load compression creep unit. The sample length was measured by measuring the displacement of the load ram with two dial gages and correcting for the bending of the apparatus caused by the applied load. In order to maintain a constant stress on the sample, the load was adjusted at 1% intervals of engineering strain by adding predetermined load increments.

Many of the tests were conducted at two stress levels, a technique first used in determing the stress dependence of creep in pure aluminum (8). The stress was applied until the steady state creep rate was determined. Then the stress was changed and maintained until the steady state creep rate was determined for the second stress. A representative creep curve is shown in figure 1. The tests were conducted in an atmosphere of dry helium.

Microstructure Analysis

The sodium chloride samples were polished by rubbing them first on wet velvet or silk and then on tracing paper smeared with one micron diamond paste. The samples were etched with glacial acetic acid plus ferric chloride. A sample showing both grains and subgrains is shown in figure 2.

Grain sizes were determined by a straight line intercept technique through the microscope. Subgrain sizes were determined with a closed ring intercept technique applied to a photograph of each sample.

Summary of Test Results

Stress, temperature, creep rates, grain sizes and subgrain sizes are presented in Table I. A plot of log steady state creep rate versus log stress is shown in figure 3. The activation energy for creep was determined from this plot to be 73.5 Kcal/mole at 160 psi. The average stress exponent determined from this plot was 7.0, while that determined from individual stress-change tests was 6.9.

Large amounts of grain growth occurred during the creep test procedure. The final grain sizes were about 0.3 mm for 512°C tests, and 1-3 mm for the 740°C tests. The regular behavior of the creep curves, however, implies that the bulk of the grain growth had occurred while the samples were coming to temperature and before the actual creep testing was started. No abrupt changes in creep rate, such as those connected with abnormal grain growth during creep, were observed. It seems possible that grain size has a small effect on creep rate in this case. In metals creeping by dislocation climb mechanism, the creep rate is related to the subgrain size, rather than the grain size. Subgrain size as a function of stress for NaCl and several metals is shown in figure 4.

TABLE I. Steady state creep rate, stress exponent, grain size and subgrain size as a function of stress and temperature.

Test No.	Sample No.	Temp. (oC)	Stress (psi)	Creep Rate (sec^{-1})	Stress Exponent, n	Final Grain Size, μ	Subgrain Size, μ
4	2.1.2	512	365	8.1×10^{-6}	7.3		
			305	2.2×10^{-6}		300	76
6	2.1.6	512	1200→800 (850 avg.)	$\sim 2.5 \times 10^{-3}$		700	20
7	2.1.1	512	470	1.8×10^{-5}	7.6		
			200	2.8×10^{-8}		500	61
10	2.1.3	740	140	1.9×10^{-4}	4.8		
			70	7.0×10^{-6}		2000	138
11	2.1.7	740	38.5	2.2×10^{-8}	8.0		
			133	4.8×10^{-4}		3000	-

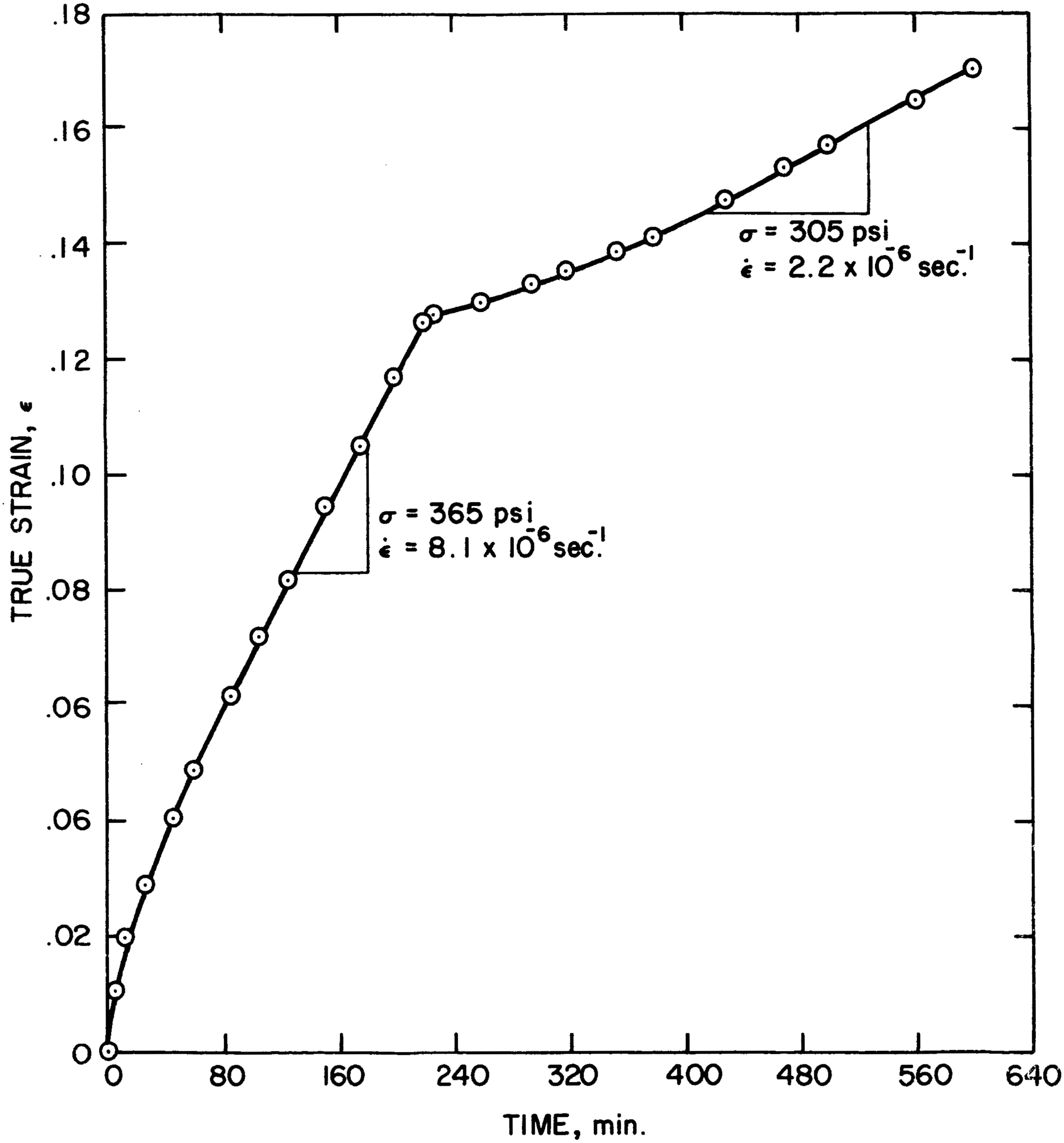

Figure 1. Representative creep curve for polycrystalline sodium chloride tested at 512°C.

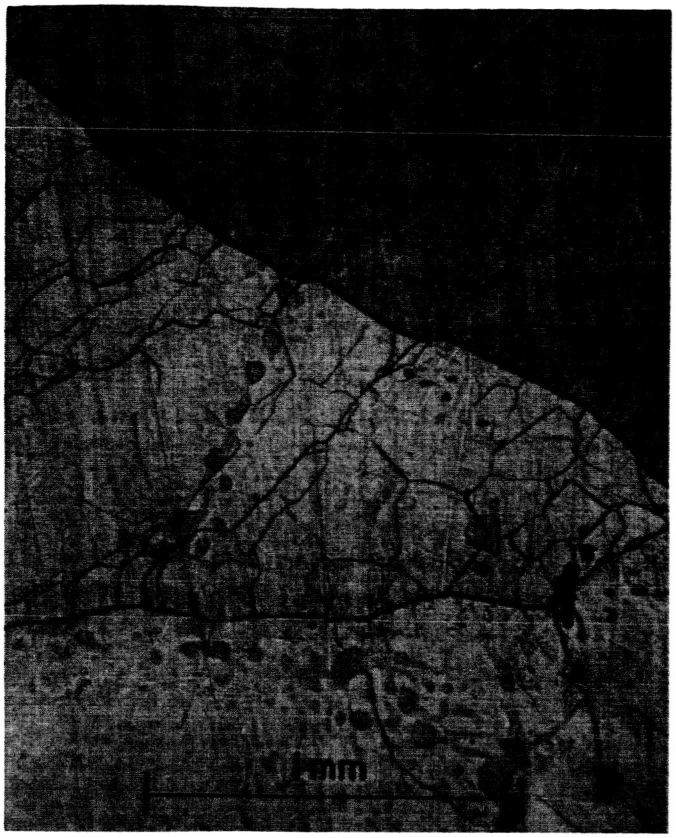

Figure 2. Microstructure of sodium chloride tested at 740°C and 70 psi final stress.

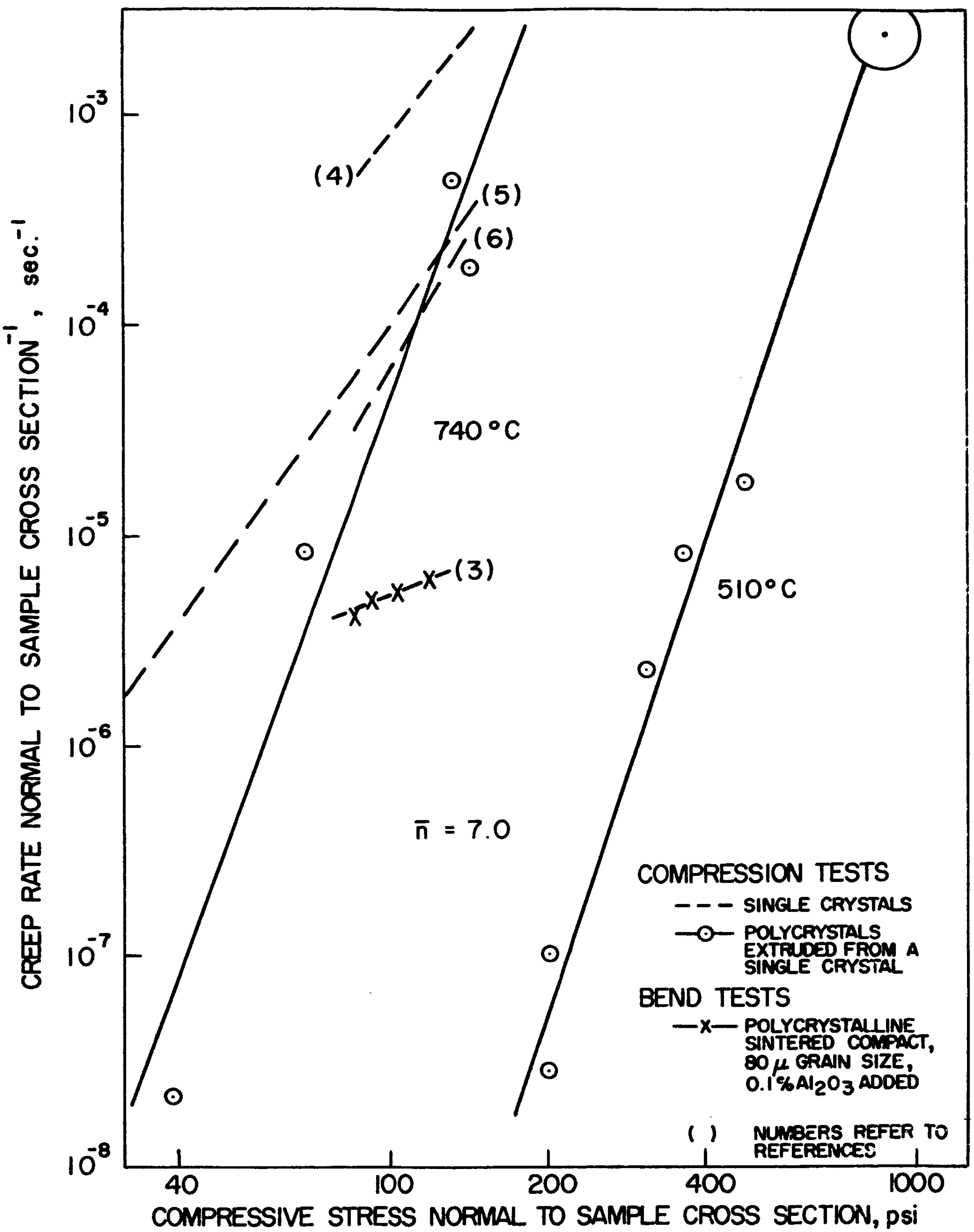

Figure 3. Plot of log steady state creep rate versus log stress for single and polycrystalline sodium chloride.

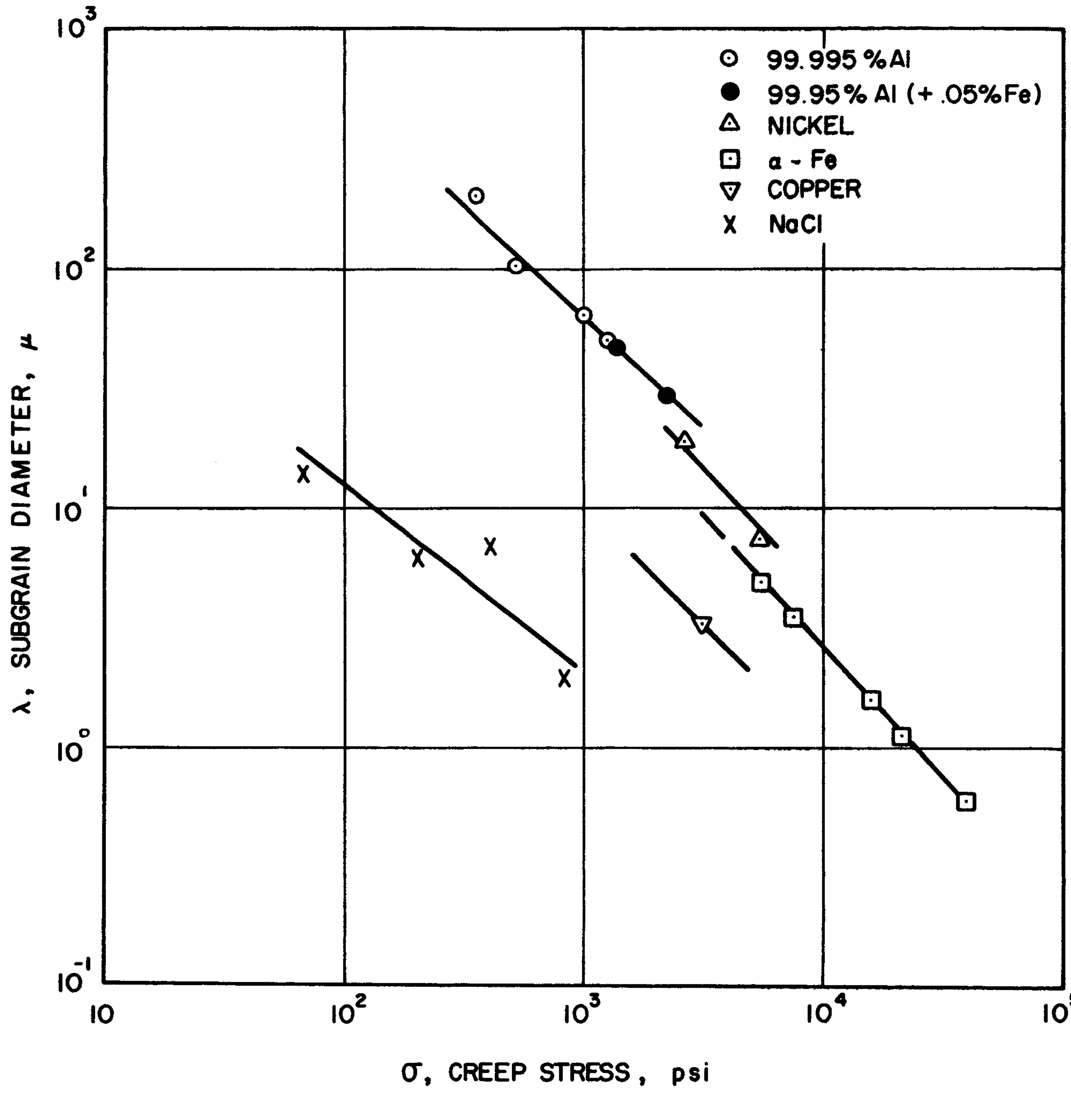

Figure 4. Influence of creep stress on the subgrain size developed during steady state creep for several pure polycrystalline metals (10, 11, 12, 13) and sodium chloride.

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